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(54) Title: FLOOR COVERING WITH BITUMEN BACKING LAYER

(57) Abstract

A floor covering having a bitumen composition as a backing layer, such as carpet tiles, which bitumen composition comprises straight run bitumen and a mixture of high density and low density polyethylene polymers blended and dispersed in the straight run bitumen as a continuous phase in an amount of up to about 10 % by weight and which bitumen composition contains a minimum of about 40 % by weight of a mineral filler material, such as limestone, and the bitumen composition having improved dimensional stability, adhesion, static loading and electrical conductivity and having lower cost and processing advantages.

bitumen & 4 layer
face fabric
latex
bitumen
2nd backing

10:32:41

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DESCRIPTION

FLOOR COVERING WITH BITUMEN BACKING LAYER

Background of the Invention

Oxidized and polymer-modified bitumens are frequently used as a backing material in the manufacture of carpet tiles. The construction of these tiles are fairly complex and may consist of a fibrous, e.g. tufted, primary cloth which has been impregnated with a cured latex to stabilize the tufting, and laminated to a secondary backing cloth using the aforementioned bitumen.

In this application, severe physical demands are placed on the bitumen to control stress relaxation in the primary tufting cloth, to maintain and retain critical physical dimensions over a wide range of possible application conditions; to prevent excessive build up of static electricity which may damage electrical and computer hardware as well as physical discomfort to building occupants; and to maintain physical dimensions under static loading conditions such as would occur when heavy objects are placed on carpet tiles over an extended period of time.

The bitumen used must also possess characteristics in the molten phase which will allow easy preparation and processing, i.e. a suitable viscosity at application temperatures with maximum thermal stability and minimum dimensional instability when applied to the manufactured product.

It has been proposed in U.K. patent application GB 2 219 802 A (Vulcanite Ltd.) to incorporate a low density polyethylene into bitumen for roofing and carpet tile backing purposes. It has been found that such blends do not fulfill all of the demands placed on a bitumen used in the context of a carpet tile backing. The static loading and dimensional stability of a carpet tile produced using such a bitumen would not be commercially acceptable and would fall outside the standard requirements laid down by the carpet tile and floor laminating industry.

Self-adhesive bitumen compositions containing polyethylene or crystalline polypropylene have been suggested for use in carpet tiles with self-adhesive backing in GB Patent 1 417 571 (Ruberoid Ltd.). Further, carpet tiles have been prepared employing a blend of bitumen and a minor amount of a thermoplastic styrene-butadiene-styrene block copolymer (SBS) as the integral backing layer in U.S. Patent 4,201,812 (Shell Oil Company). However, the block copolymer presents processing, compatibility, cost and other difficulties in its used as a carpet backing blend.

SUMMARY OF THE INVENTION

This invention relates to bitumen compositions having improved dimensional stability, adhesion, static loading and electrical conductivity characteristics, particularly, but not exclusively, such compositions for use in the production of carpet tiles, flooring and other laminating applications.

According to the present invention, there is provided a floor covering having a bitumen composition as a backing layer which fulfills all of the demands described earlier and also provides a considerable cost saving advantage over systems described earlier and already in operation.

Thus the present invention provides a floor covering having a bitumen composition backing layer, which bitumen composition comprises:

a) a dispersed phase of straight run bitumen having a penetration value of up to 100 dmm;

b) a continuous phase of a mixture of low density polyethylene and a high density polyethylene;

the bitumen composition having a softening point of 115°C to 128°C and a penetration value of 10 to 15 dmm.

In one embodiment, this invention relates to a bitumen polymer filler blend suitable for use as a flooring of a laminate backing layer. The blend comprises a minimum inert filler material content of about 40% w/w of the final

blend. The bituminous component comprises a straight run bitumen of high asphaltene and resin content with a penetration value (IP 49/83) of 100 decimillimeters (dmm) or less and a blend containing a mixture of low density and high density polyethylenes. Generally, the total polyethylene content does not exceed about 10% w/w of the final blend.

The present invention provides a bitumen composition useful as a backing layer for carpet tiles which composition is low cost and can be manufactured from locally available materials, that is, straight run bitumen, rather than blown or oxidized bitumen, and either virgin or preferably low cost scrap or recycled polyethylene as a modifier. The bitumen-polyethylene blends employed as a backing layer provide carpet tiles of improved dimensional stability and better electrical conductivity so that an antistatic agent is not required to meet existing IBM/ICL standards for conductivity.

In addition, the bitumen-polyethylene composition provides better adhesion due to the increased tackiness of the bitumen composition, so that there is an improved quality of lamination between the bitumen backing layer and the back surface of the latex-coated and cured, primary, needle-punched or tufted backing sheet. The bitumen composition permits a reduction in the application or coating temperature, resulting in energy savings and permitting the use of less expensive, lower melting point secondary backing sheets on the back surface of the tile, for example, the use of non-woven polypropylene sheets in place of higher melting point polyester or polyester-polypropylene backing sheets. The carpet tile produced has a fibrous face surface and a back surface integrally bonded to the bitumen composition as a backing layer and typically a secondary backing sheet secured to the back surface of the bitumen backing layer.

In comparison to the use of styrene-butadiene-

styrene block copolymers, there are no problems relating to bitumen compatibility, the bitumen is not temperature sensitive, does not cross link in prolonged storage at high temperature and does not require a special bitumen composition with hazardous components.

The bitumen-polyethylene blend composition used in the invention should have a softening point of 115°C to 128°C, e.g. 118°C to 124°C, and a penetration of 10 dmm to 15 dmm, e.g. 10 dmm to 12 dmm. The softening point (ring and bill test) and penetration values are measured in accordance with the Institute of Petroleum specifications, respectively IP 58/83 and IP 49/83. The bitumen composition is prepared by heating the bitumen to about 180°C to 200°C, e.g. 190°C, and the polyethylene polymer is then added typically with the high density polyethylene added first and the polyethylene polymer mixed with continuous agitation with moderate shear for sufficient time, e.g. over 60 minutes, until a smooth, homogeneous dispersion is obtained, that is, until the bitumen is dispersed as a dispersed phase in a continuous phase of the polyethylene. Generally, the quality of the dispersion may be monitored wherein a sample of the blend is viewed under ultraviolet illumination to affect fluorescence of the polymer so the quality of the blend can be visually assessed.

The bitumen composition is employed as a backing layer for floor laminates, particularly carpet tiles by heating the bitumen composition and applying one or more coating layers to the back surface of a fibrous face floor surface covering. Generally, the bitumen composition is applied at a temperature of 160°C to 180°C, e.g. 165°C to 170°C, at the coating or application station which is lower than normal coating temperature with blown bitumen and at a viscosity of 6,000 cps to 35,000 cps 6 to 35 pascal seconds), e.g. 20,000 cps to 35,000 cps (20 to 35 pascal seconds). Generally, SBS-modified bitumens are applied as backing layers at temperatures of 180°C to 190°C; however,

at lower temperatures of 160°C to 170°C and less, the viscosity of the high density polyethylene (HDPE) and low density polyethylene (LDPE) modified straight run bitumen does not increase as rapidly as the viscosity of blown or oxidized bitumen, so that application at lower temperatures with resulting savings in energy cost and the use of less expensive secondary backing are obtained. The lower application temperature permits the use of low melting point, 160°C to 170°C, 100% polypropylene, non-woven or other sheet material as a secondary backing. The bitumen may be applied in any manner, such as by a lick roller or a lay-in technique.

The bitumen composition employs straight run bitumen of high asphaltene and resin content and which has a penetration of 100 dmm or less, typically less than about 60 dmm, e.g. 20 dmm to 60 dmm particularly 40 to 60 dmm. Straight run bitumen is more readily available and of lower cost and lower viscosity than blown bitumen. Generally, the bitumen consists all of straight run bitumen; however, very minor amounts, for example, up to 10% by weight of other bitumens, such as blown bitumen or other hydrocarbon products, can be incorporated if desired.

The straight run bitumen is blended with a mixture of high density and low density polyethylene polymers in an amount sufficient to form a dominant, continuous phase of the polyethylene polymer. Once, the polyethylene concentration has reached the phase transition level, further polyethylene polymer addition does not increase the softening point of the bitumen blend. Generally, the polyethylene is employed in total amounts of up to about 12% by weight, e.g. between 8% to 10%. For example, the HDPE may range from about 0.5% to 3.0% by weight, such as 1% to 2%, while the LDPE may range from about 3.0% to 9.5% by weight, such as 3% to 6%. It has been found that the use of a moderate density polyethylene to reduce the softening point of the straight run bitumen is not satisfactory due to

the large concentrations required, and that processing and cost factors make its use unsatisfactory. The use of low density polyethylene alone is also not satisfactory to produce a commercially acceptable bitumen backing for carpet tile of defined penetration value and softening point.

The properties of polyethylenes are dependent upon their molecular configurations, molecular weights and molecular weight distributions. An important factor is the selection and blending of the polyethylenes used. Commercially, polyethylenes are graded in terms of the density and the melt flow index, with both measurements necessary to define any particular grade. The melt flow index (MFI) is the number of grams of a polymer that can be forced through a 0.0825-inch (2.1 mm) orifice in 10 minutes at 190°C by a pressure of 2,160 grams (ASTM D1238-70).

Typically, the high density polyethylene component should have a density between about 0.945 g/m^3 and 0.97 g/m^3, and a melt flow index greater than about 0.2, such as 0.2 to 10, e.g. 4 to 6. The low density component of this invention should have a density of about between 0.915 g/m^3 and 0.93 g/m^3, and a melt flow index greater than about 0.2, such as 2 to 10.

Generally, the bitumen composition includes an inert mineral filler material to reduce cost and to prevent cold flow of the composition in use, such as the use of slate dust or limestone, in amounts of greater than about 40% by weight, such as between about 40% to 65% by weight, and typically 50% to 55%. The bitumen composition may contain a wide variety of various modifiers and additives commonly used in carpet tile backing layers, provided such modifiers and additives are not detrimental to the accepted quality of the backing layer. Such modifiers and additives may include, but not be limited to: antistatic agents, such as fatty amines; and carbon black, including master batch mixtures of polyethylene and carbon black, for example, HDPE and LDPE with carbon black used for incorporating into the

bitumen blend; other fillers; pigments for color; and minor amounts of other polymers and additives.

The invention will be described for the purposes of illustration only in connection with certain embodiments; however, it is recognized that various changes, modifications, additions and improvements may be made to the invention as illustrated by a person skilled in the art, all falling within the spirit and scope of the invention.

Description of the Embodiments

A Hubis mixer was charged with 311 Kg of 50 penetration (IP 49/83) straight run bitumen at 190°C, and 28 Kgs of LDPE (MFI 3, density 0.95 g/m^3), and 10.5 Kgs of HDPE (MFI 4, density 0.92 g/m^3) were added, and the mixing cycle started. After 10 minutes, a sample of the blend was removed for ultraviolet examination of assess blend quality. The mixing was then continued in 10-minute cycles until no further improvement in the quality of the dispersion could be observed. When this point was reached, 350 Kgs of limestone filler was added to produce a final batch weight of 700 Kgs.

The batch was used to manufacture sample tiles employing a lick roller to apply the bitumen to the back surface of a flooring substrate with the bitumen temperature at the application point of 153°C to 162°C. The polyethylene-bitumen composition had a softening point of 121°C (IP 58/83), a penetration of 10 dmm (IP 49/83), and a viscosity temperature profile as follows:

Viscosity (pascal seconds)	Temperature (°C)
47.0	130
30.0	140
20.0	150
14.0	160
9.5	170
7.5	180
6.0	190

Dispersion Quality - It was found that the best obtainable dispersion was produced after 20 to 30 minutes,

using the high speed disintegrator. Carpet tiles so manufactured were compared with similar commercial carpet tiles known as Collage (a trademark of Interface Flooring Systems, Inc.) as a control tile and made in the same manner, except with blown bitumen applied at 190°C.

Full Scale Industrial Trial

Results	Collage Control Tile	Polymer Modified Tile
Aachen Test		
Change in dimensions after 24 hours conditioning	Wa.1 N.A. Wa.2 N.A. Wt.1 N.A. Wt.2 N.A.	Wa.1 -.02 Wa.2 -.04 Wt.1 +.02 Wt.2 -.02
Change in dimensions after 2 hours at 60°C	Wa.1 -.106 Wa.2 -.121 Wt.1 -.029 Wt.1 -.016	Wa.1 -.130 Wa.2 -.016 Wt.1 -.012 Wt.2 -.022
Change in dimensions after 2 hours water at 20°C	Wa.1 -.068 Wa.2 -.084 Wt.1 .001 Wt.2 .002	Wa.1 -.063 Wa.2 -.020 Wt.1 .033 Wt.2 .031
Change in dimensions after 24 hours at 60°C	Wa.1 -.230 Wa.2 -.244 Wt.1 -.140 Wt.2 -.072	Wa.1 -.166 Wa.2 -.186 Wt.2 -.014 Wt.2 -.056
Change in dimensions after 48 hours reconditioning at 20°C, 65% RH	Wa.1 -.266 Wa.2 -.228 Wt.1 -.117 Wt.2 -.068	Wa.1 -.126 Wa.2 -.178 Wt.1 0 Wt.2 -.034

Limitations of +0.2% to -0.4% are generally acceptable, except for U.S.A. and France, -0.1% to -0.2%.

Tuft Withdrawal Test

	Control Tile	Polymer Modified Tile
Mean force required ti renive tuft	4.26 Newtons	4.40 Newtons

Static Conductivity Test

Conditions: 19.9°C, 29% Relative Humidity

	Control Tile	Polymer Modified Tile
IBM	1.2x10*11 ohms	7.75x10*10 ohms
ICL	1.2x10*11 ohms	2.9x10*11 ohms

Adhesion/Delamination Test

Again the adhesion within the polymer modified tile provide to be superior to that exhibited within the oxidized tile.

The low and high density polyethylene modified straight run bitumen blend provides a unique bitumen backing for floor laminates and provides energy saving, processing and manufacturing advantages.

CLAIMS

1. A floor covering having a bitumen composition backing layer, which bitumen composition comprises:

a) a dispersed phase of straight run bitumen having a penetration value of up to 100 dmm;

b) a continuous phase of a mixture of low density polyethylene and a high density polyethylene;

the bitumen composition having a softening point of 115°C to 128°C and a penetration value of 10 to 15 dmm.

2. A floor covering as set forth in claim 1 wherein said composition also comprises up to 10 percent by weight of the composition of blown bitumen as a dispersed phase.

3. The floor covering as set forth in claim 1 or claim 2 wherein said composition also comprises at least 40 percent by weight of the composition of a mineral filler material.

4. The floor covering as set forth in claim 1, 2 or 3 wherein the composition contains from 3 percent to 9.5 percent by weight of the composition of high density polyethylene and from 0.5 percent to 3.0 percent by weight of the composition of a low density polyethylene.

5. A floor covering of any of the preceding claims wherein the high density polyethylene has a density of from 0.945 gm/cm^3 to 0.970 g/cm^3, and the low density polyethylene has a density of from 0.915 g/cm^3 to 0.930 g/cm^3.

6. A floor covering of any one of the preceding claims wherein the composition contains from 4 percent to 10 percent by weight of the composition of the mixture of high density polyethylene and low density polyethylene.

7. A floor covering of any one of the preceding claims wherein the penetration value of the straight run bitumen is from 40 dmm to 60 dmm.

8. A floor covering of any one of the preceding claims wherein the viscosity of the bitumen composition is from 6 pascal seconds to 30 pascal seconds.

9. A floor covering of any one of the preceding claims wherein the high and low density polyethylene have a melt flow index of 0.2 to 10.

10. A floor covering of any one of the preceding claims wherein the floor covering comprises a carpet tile having a fibrous face surface and a back surface, the bitumen composition being bonded to the back surface.

11. A floor covering as set forth in claim 10 which includes a non-woven polypropylene secondary backing sheet bonded to the back surface of the bitumen composition.

12. A floor covering of any one of the preceding claims wherein the bitumen composition has a penetration value of 10 dmm to 12 dmm and a softening point of 118°C to 124°C.

13. A bitumen blend suitable for use in carpet tiles comprising a straight run bitumen having a penetration of 100 dmm or less; a high density polyethylene having a density between 0.945 g/cm^3 and 0.970 g/cm^3 and a melt flow index of greater than 0.2, the concentration of the high density polyethylene component being between 0.5 percent and 3 percent by weight of the total blend, a low density polyethylene having a density of between 0.915 g/cm^3 and 0.930 g/cm^3 and a melt flow index greater than 2, the concentration of the low density polyethylene component being between 3 percent and 9.5 percent by weight of the total blend; and an inert mineral filler which comprises at least 40 percent by weight of the total blend.

INTERNATIONAL SEARCH REPORT

PCT/GB 91/00553

International Application N·

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

Int.Cl. 5 D06N7/00 ; C08L95/00

II. FIELDS SEARCHED

Minimum Documentation Searched[7]

Classification System	Classification Symbols
Int.Cl. 5	D06N ; C08L

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched[8]

III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

Category *	Citation of Document,[11] with indication, where appropriate, of the relevant passages[12]	Relevant to Claim No.[13]
Y	DATABASE JAPS/JPO & JP-A-1123862 (FUJI KOSAN) 16 May 1989 see abstract	1, 2, 6, 10
Y	US,A,3853800 (P. HABERL) 10 December 1974 see column 7, lines 1 - 3; claims 1-6; see column 1, line 66 - column 2, line 7	1, 2, 6, 10
Y	GB,A,2219802 (VULCANITE) 20 December 1989 see page 2, lines 16 - 36; claims 1, 2 (cited in the application)	1, 10
A	EP,A,299700 (EXXON RESEARCH) 18 January 1989 see page 4, lines 37 - 40; claims 1, 3, 8 see page 1, lines 3 - 4	1, 2, 4-6, 13
A	DE,A,3527525 (SCHERING) 05 February 1987 see page 3, lines 47 - 59; claims 1-4, 8	1

* Special categories of cited documents :[10]

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IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
08 AUGUST 1991	20. 08. 91
International Searching Authority	**Signature of Authorized Officer**
EUROPEAN PATENT OFFICE	PFANNENSTEIN H.

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ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.

GB 9100553
SA 46375

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A-3853800	10-12-74	NL-A- 7400215	09-07-74
GB-A-2219802	20-12-89	GB-A- 2223229	04-04-90
EP-A-299700	18-01-89	US-A- 4868233	19-09-89
		AU-B- 601296	06-09-90
		AU-A- 1903188	19-01-89
		JP-A- 1103664	20-04-89
DE-A-3527525	05-02-87	None	

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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82